

TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371		Attorney Docket No. <u>01115</u>
INTERNATIONAL APPLICATION NO. PCT/FR99/03184		INTERNATIONAL FILING DATE December 17, 1999
TITLE OF INVENTION IMPROVED METHOD FOR FILTERING A METAL LIQUID ON A BED OF REFRACTORY PARTICULATE MATERIAL		U.S. Application No. (if known, see 37 CFR 1.51) 09/856460
APPLICANT(S) FOR DO/EO/US Herve Lescuyer and Alain Dubus		PRIORITY DATE CLAIMED December 21, 1998
Applicant herewith submits to the United States Designated Office (DO/EO/US) the following items and other information:		
<ol style="list-style-type: none"> 1. <input checked="" type="checkbox"/> This is a FIRST submission of items concerning a filing under 35 U.S.C. 371. 2. <input type="checkbox"/> This is a SECOND or SUBSEQUENT submission of items concerning a filing under 35 U.S.C. 371. 3. <input type="checkbox"/> This is an express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(1). 4. <input checked="" type="checkbox"/> A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date. 5. <input checked="" type="checkbox"/> A copy of the International Application as filed (35 U.S.C. 371(c)(2)) <ol style="list-style-type: none"> a. <input checked="" type="checkbox"/> is transmitted herewith (required only if not transmitted by the International Bureau). b. <input checked="" type="checkbox"/> has been transmitted by the International Bureau. c. <input type="checkbox"/> is not required, as the application was filed in the United States Receiving Office (RO/US). 6. <input checked="" type="checkbox"/> A translation of the International Application into English (35 U.S.C. 371(c)(2)). 7. <input type="checkbox"/> Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3)). <ol style="list-style-type: none"> a. <input type="checkbox"/> are transmitted herewith (only if not required by the International Bureau). b. <input type="checkbox"/> have been transmitted by the International Bureau. c. <input type="checkbox"/> have not been made; however, the time limit for making such amendments has NOT expired. d. <input type="checkbox"/> have not been made and will not be made. 8. <input type="checkbox"/> A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)). 9. <input type="checkbox"/> An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)). 10. <input type="checkbox"/> A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)). 		
Items 11 to 16 below concern document(s) or information included:		
<ol style="list-style-type: none"> 11. <input checked="" type="checkbox"/> An Information Disclosure Statement under 37 CFR 1.97 and 1.98. 12. <input type="checkbox"/> As assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included. 13. <input checked="" type="checkbox"/> A FIRST preliminary amendment. 14. <input type="checkbox"/> A SECOND or SUBSEQUENT preliminary amendment. 15. <input type="checkbox"/> A substitute specification. 16. <input type="checkbox"/> A change of power of attorney and/or address letter. 16. <input checked="" type="checkbox"/> Other items or information: Application Data Sheet 		




23338

PATENT TRADEMARK OFFICE

17. <input checked="" type="checkbox"/> The following fees are submitted:				CALCULATIONS PTO USE ONLY	
BASIC NATIONAL FEE (37 CFR 1.492 (a)(1)-(5):				J010 Rec'd PCT/TTG 07 JUN 2001	
Neither international preliminary examination fee (37 CFR 1.482)					
Nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO					
And International Search Report not prepared by EPO or JPO..... \$1,000.00					
International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by EPO or JPO.....\$860.00					
International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International search fee (37 CFR 1.445(a)(2)) paid to USPTO..... \$710.00					
International preliminary examination fee paid to USPTO (37 CFR 1.482) But all claims did not satisfy provisions of PCT Article 33(1)-(4).....\$690.00					
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Surcharge of \$130.00 for furnishing oath or declaration later than <input type="checkbox"/> 20 <input checked="" type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(e)).				\$130.00	
CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE		
Total Claims	10 -20=		X \$18.00	\$	
Independent Claims	1 -3=		X \$80.00	\$	
MULTIPLE DEPENDENT CLAIM(S) (if applicable)				\$	
TOTAL OF ABOVE CALCULATIONS =				\$990.00	
Reduction of 1/2 for filing by small entity, if applicable. A Small Entity Statement must also be filed (Note 37 CFR 1.9, 1.27, 1.28).				\$	
SUBTOTAL =				\$990.00	
Processing fee of \$130.00 for furnishing English translation later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(f)).				\$	
TOTAL NATIONAL FEE =				\$990.00	
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31).				\$	
TOTAL FEES ENCLOSED =				\$990.00	
				Amount to be refunded:	\$
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- a. ☐ A check in the amount of \$ _____ to cover the above fees is enclosed.
- b. ☐ Please charge my Deposit Account No. 04-0753 in the amount of \$ _____ to cover the above fees. A duplicate copy of this sheet is enclosed.
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Dkt. 01115

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:

HERVE LESCUYER et al

PCT

Serial No.: none assigned
(PCT/FR99/03184)

Filed: Concurrently Herewith

For: IMPROVED METHOD FOR FILTERING A METAL LIQUID
ON A BED OF REFRACTORY PARTICULATE MATERIAL

PRELIMINARY AMENDMENT AND INFORMATION DISCLOSURE STATEMENT

Honorable Commissioner of Patents and Trademarks
Washington, D.C. 20231

Sir:

Before calculation of the filing fee, please amend
the above-identified application as follows:

IN THE ABSTRACT:

Please add the following Abstract of the Disclosure:

--ABSTRACT OF THE DISCLOSURE

A filtration method for liquid metal in which the metal liquid metal on a bed of refractory particulate material having an open porosity between 5 and 30%. Electrofused corundum may be used as the refractory particulate material. The invention enable the use of reduced retention times while maintaining a quality level at least equal to that obtained with other particulate materials.--

IN THE SPECIFICATION:

Page 1, line 1: Field of the invention;
line 6: DESCRIPTION OF RELATED ART.

Page 2, line 11: SUMMARY OF THE INVENTION.

Page 5, between lines 31 and 32, insert:

--BRIEF DESCRIPTION OF THE DRAWINGS

Figures 1a and 1b are graphs of pore distributions for the prior art and an example of the invention; and

Figure 2 is a graph of filtration efficiency versus residence time for filters according to the prior art and an example of the invention.

DESCRIPTION OF THE PREFERRED EMBODIMENTS--

IN THE CLAIMS:

Page 10, above line 1: What is claimed is:

Please enter the following amended claims as set forth below and in the attached Appendix:

1. (Amended) A filtration method for liquid metal comprising passing said liquid metal on a bed of refractory particulate material having an open porosity between 5 and 30%.

2. (Amended) The filtration method according to claim 1, wherein the liquid metal has a residence time in the

particulate material bed greater than 1 sec and less than 500 secs.

3. (Amended) The filtration method according to claim 1, wherein the porosity substantially stems from pores with a diameter greater than 10 μm .

4. (Amended) The filtration method according to claim 1, wherein the material has a particle size between 0.2 and 20 mm and the bed has a thickness between 4 and 40 cm.

5. (Amended) The filtration method according to claim 1, wherein the material is electrofused corundum.

6. (Amended) The filtration method according to claim 1, wherein the liquid metal is selected from the group consisting of aluminum, magnesium and alloys thereof.

7. (Amended) The filtration method according to claim 5, wherein the corundum is obtained by method steps comprising electrofusion of alumina, a casting, a cooling and solidification in order to obtain said porosity, a crushing, then a screening process.

8. (Amended) A corundum used in the method according to claim 5, having a porosity between 5 and 30%.

9. (Amended) A filtration device for liquid metal including the corundum according to claim 8.

--11. (New) The filtration method according to claim 3, wherein the porosity substantially stems from pores with a diameter between 10 and 200 μm .--

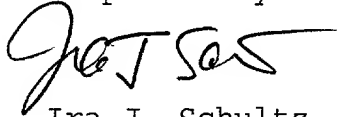
Please cancel Claim 10 without prejudice or disclaimer of the subject matter thereof.

REMARKS

The claims have been amended to delete all multiple dependencies and to place the claims in generally better form for U.S. practice.

Attached hereto is the Search Report of the corresponding French application, together with copies of references cited therein, which are listed on the attached Form PTO-1449.

Respectfully submitted,



Ira J. Schultz
Registration No. 28666

APPENDIX

Page 1, line 1: [Technical field] Field of the invention;

line 6: [State of the art] DESCRIPTION OF RELATED ART.

Page 2, line 11: [Description of the invention] SUMMARY OF THE INVENTION.

Page 10, above line 1: [CLAIMS] What is claimed is:

1. (Amended) A filtration method for liquid metal [by having] comprising passing said liquid metal [pass] on a bed of refractory particulate material [characterized in that the particulate material has] having an open porosity between 5 and 30%.

2. (Amended) The filtration method according to claim 1, [characterized in that the residence time of] wherein the liquid metal has a residence time in the particulate material bed [is] greater than 1 sec and less than 500 secs.

3. (Amended) The filtration method according to [any of claims 1 or 2, characterized in that] claim 1, wherein the porosity substantially stems from pores with a diameter greater than 10 μm [and preferably between 10 and 200 μm].

4. (Amended) The filtration method according to [any of claims 1 to 3, characterized in that] claim 1, wherein the material has a particle size between 0.2 and 20 mm and the bed has a thickness between 4 and 40 cm.

5. (Amended) The filtration method according to [any of claims 1 to 4, characterized in that] claim 1, wherein the material is electrofused corundum.

6. (Amended) The filtration method according to [any of claims 1 to 5, characterized in that] claim 1, wherein the liquid metal is selected from the group consisting of [aluminium] aluminum, magnesium [or their] and alloys thereof.

7. (Amended) The filtration method [for obtaining corundum] according to claim 5, [characterized in that it comprises] wherein the corundum is obtained by method steps comprising electrofusion of alumina, a casting, a cooling and solidification in order to obtain said porosity, a crushing, then a screening process.

8. (Amended) A corundum used in the method according to [any of claims 1 to 6, or obtained according to the method of] claim [7] 5, [characterized in that it has] having a porosity between 5 and 30%.

9. (Amended) A filtration device for liquid metal including the [material] corundum according to claim 8.

IMPROVED METHOD FOR FILTERING A LIQUID METAL ON A
BED OF REFRACTORY PARTICULATE MATERIAL

Technical field

The invention relates to an improved method for filtering a liquid metal, in particular aluminium, magnesium, or their alloys on a thick bed of refractory gravel.

5

State of the art

The filtering of liquid aluminium on a thick bed of gravel made of sintered alumina, so-called tabular alumina which is an alpha alumina, generally in the form of beads or crushed grains, is known, for removing solid or liquid inclusions from it. It is very important to be able to improve this removal in particular when aluminium is used for obtaining very thin sheets in order to reduce the risk of waste material; indeed, the thinner the produced sheet, the more it becomes necessary to remove the small sized inclusions, in addition to the large ones, because they produce defects which become detrimental.

The bed of sintered alumina gravel generally has a thickness of the order of 40 cm. The purification rate of liquid metal after filtering inclusions by this type of alumina is limited; thus said metal may further contain after filtration up to 10,000 particles of a size greater than 20 μm per kg, even for a residence time which generally is between 100 and 500 secs, wherein said purification rate is very variable depending on the size of the particles and from one casting operation to another. Furthermore, it should be noted that this type of alumina is expensive.

The applicant has thus tried to reduce the amount of inclusions present in the filtered liquid metal by more particularly concerning herself with improving the removal of small size inclusions. She has also tried to reduce the cost, and more generally improve the performances of the liquid metal filtering process through a bed of particulate material, while trying to find a solution to the problem of recycling said bed of particulate material.

Description of the invention

The invention is a method for filtering a liquid metal wherein said liquid metal flows through a thick bed of refractory particulate material having an open porosity between 5 and 30%.

This method is essentially applied to aluminium, magnesium or their alloys.

Porosity, which corresponds to the porous volume of the grains of the bed (surface porosities and internal porosities) is measured by mercury porosimetry; it is due to pores of a diameter essentially greater than 10 μm and generally less than 200 μm in order to maintain good resistance to erosion. The particle size is preferably between 0,2 and 20 mm and the bed has a thickness of 4 to 40 cm. The residence time of the liquid metal in the gravel bed may be of the same order of magnitude as that used for the tabular alumina gravel but it is remarkable to note that a purification, at least equivalent, or even superior, to that obtained with said alumina, is obtained for residence times less than 200 secs, or even less than 50 secs. With such short residence times, the size of the industrial facilities may be

significantly reduced, while maintaining a same filtration efficiency.

As a comparison, tabular alumina gravel has a very low porosity generally less than 3% due to very fine pores less than 10 μm in majority.

The material used in the invention advantageously is an alumina. According to the preferred embodiment of the invention, said refractory material is an electrofused corundum obtained by fusing an alumina in an arc oven, followed by a casting process, preferably in moulds, by adjusting the cooling and solidification state in order to obtain the desired open porosity, and by a crushing and/or grinding process, for example in a roller or hammer mill, wherein the obtained gravel is then screened to the desired size and dust removed. Crystallization modifiers such as F, B, Y, MgO or SiO_2 may be added for controlling the porosity. Preferably, white corundum is used in order to prevent any risk of chemically contaminating the liquid metal.

The removal rate of the inclusions is always greater than 95%, even greater than 97%, regardless of the size of said inclusions, which also results in a significant reduction in the presence of particles with small dimensions.

It is important to note that even if it is always possible to use a long residence time in order to improve the removal rate of the inclusions, with the invention reduced residence times may be used while maintaining a quality level at least equal, or even superior to that observed with other particulate materials and a remarkable constancy from one casting process to another, which for example is not the case with tabular alumina. The possibility of filtering with

reduced residence times also results in a given thickness of the gravels, by the possibility of increasing the metal flow rate (or the filtration rate) with equal efficiency. This possibility may also limit
5 the release of inclusions during surges of the metal flow rate.

Thus, the residence time may be as reduced as 1 sec, preferably at least 2 secs, and still preferably at least equal to 5 secs; it is generally less than 500
10 secs, preferably less than 200 secs, and preferably still less than 100 secs, and is advantageously located between 2 and 200 secs or better between 2 and 100 secs, or even between 5 and 100 secs, the shortest residence times being determined by the level of the
15 desired removal rate and the accepted risk of release of the inclusions.

As an illustration, the number of particles present in the filtered metal does not generally exceed 600 particles with a size greater than 20 μm per kg of
20 filtered liquid metal, the amount of smaller particles being reduced by the same factor. It appears that not only the fixing of the inclusions is better carried out but even that no release occurs.

It seems that the presence of the required minimum
25 porosity plays a primordial role for increasing the filtration efficiency, the retention rate for the inclusions, the filtration rate, or for reducing the size of retained inclusions and for preventing their release.

30 Also, the retention capacity of the inclusions in corundum is larger than in tabular alumina, i.e., at a constant purification rate of the liquid metal, the lifetime of the filters is increased. The frequency of

operations for changing the filtering bed is then reduced, so that stops in production may also be limited.

It may also be believed that other parameters may play an important role like roughness or surface chemistry which may increase wettability by the liquid metal and the retention capacity (or adhesion) of the inclusions on the gravel. The phenomena involved may be different from those occurring with tabular alumina of very low porosity. A partly intragranular filtration may in particular occur whereas for a tabular alumina, pores of a diameter less than 10 μm are not accessible to liquid aluminium in the normal operating conditions.

The fact of being able to use high filtration rates while improving the purification rate and retention rate may increase productivity, reduce the size of filtering facilities, reduce consumption of filtering medium and thus obtain a reduction in costs all the more significant as corundum is itself less expensive than tabular alumina.

Further, corundum provides the advantage of being able to be easily recycled by re-manufacturing it through simple fusion whereas for tabular alumina, if the intention is to avoid an expensive re-manufacture, a complex regeneration treatment must be performed as it generally comprises the combination of a treatment with different aqueous solutions, which are as many effluents to be treated and/or recycled, and a regeneration heat treatment.

Said bed may optionally be reinforced in order to facilitate its handling.

The following example illustrates the invention by comparing it to the state of the art.

Two filtering beds were successively used in the same facility for performing several castings each.

The first bed, according to the prior art, is based on tabular alumina beads with a particle size of 3/6 mesh, i.e. between 3.35 mm and 6.70 mm, and with a porosity of 2.8% mostly due to pores with a diameter less than 7 μm ; it has a thickness of 40 cm. Measurement of the specific surface, according to the multi-molecular adsorption measurement method known as the BET (Brunauer, Emmet and Teller) method, gave a value of 0.012 m^2/g for this bed.

The second bed, according to the invention, is a white corundum (with a purity higher than 99.6%) with porosity of 10.7% mostly due to pores with a diameter between 10 and 250 μm , its particle size is between 3 and 6 mm and the bed has a thickness of 40 cm. It is obtained by casting liquid alumina into metal ingot moulds, wherein the cooling and solidification rate is 50 to 100°C/hr, by crushing the solidified product and then grinding it in a roller mill and by screening it between sieves with 3 and 6 mm apertures. The BET specific surface of this bed was 0.09 m^2/g . The particles of the bed were rather of an acicular shape, even needle-shaped in certain tests.

The porosity distribution of the bed's particles according to the prior art (curve A) and according to the invention (curve B) is illustrated in Fig. 1. Fig. 1a gives the porous volume V (in cm^3/g) versus the pore diameter \varnothing (in μm). Fig. 1b gives the same pore distributions as Fig. 1a in cumulative form (cumulative pore distributions).

The liquid metal used is an aluminium-magnesium alloy (1.2%) wherein known additions of inclusions with

a size less than 120 μm were made in order to obtain between 10,000 and 35,000 inclusions/kg of metal depending on the castings. The residence time of liquid aluminium in the filtering bed is 100 secs during each of the castings.

Counting of the inclusions is carried out by means of a LiMCA (Liquid Metal Cleanliness Analysis) apparatus marketed by BOMEM and implementing in liquid aluminium the well-known counting method of the so-called Counter Coulter type which measures both number and size of the particles by measuring electric resistance when the latter pass through a calibrated port.

The tables below give for each casting, the inclusion removal rate, in %, observed during the casting depending on the size of the inclusions. Table 1 corresponds to tests according to the prior art, Table 2 corresponds to the tests according to the invention.

Table 1: Inclusion removal rate after filtration on a tabular alumina bed (comparative tests)

Inclusion size μm	20-40	40-60	60-80	>80
Casting 1	77	73	77	87
Casting 2	95	93	91	94
Casting 5	88	90	87	92
Casting 6	84	90	92	98
Average	86	87	87	93

It is seen that in these tests, dispersion of the results is significant both from one casting to another and depending on the particle size and that on average,

removal rate is insufficient as it does not exceed 93% for the largest inclusions. The randomness of the inclusion removal rate is particularly detrimental because it considerably increases the risk of waste material when thin or very thin metal sheets are obtained subsequently.

Table 2: Inclusion removal rate after filtration on a corundum bed according to the invention

Inclusion size μm	20-40	40-60	60-80	80-100
Casting 1	98	99	98	97
Casting 2	99	99	100	99
Casting 3	98	98	96	99
Casting 4	99	99	99	98
Casting 5	99	99	98	97
Average	99	99	98	98

It is observed that the filtering result is both excellent in homogeneity and in level as the removal rate is on average at least 98%. In particular, the smallest particles have been very well removed.

Fig. 2, which gives the filtration efficiency E (in %) versus the residence time T (in seconds) for a filter according to the prior art (curve A) and according to the invention (curve B), shows that filtering beds according to the invention maintain a very high filtering efficiency for residence times less than 200 secs, whereas the efficiency of filtering beds of the prior art are substantially reduced for residence times less than 200 secs. The residence time corresponds to the equivalent drum-vacuum filtering

rate, i.e. it corresponds to the minimum residence time
calculated from the metal flow rate as if it were a
laminar flow. In spite of a close, even lower specific
surface than that of the bed of the prior art, the bed
5 according to the invention did exhibit larger
filtration efficiency.

CLAIMS

1. A filtration method for liquid metal by having said liquid metal pass on a bed of refractory particulate material characterized in that the particulate material has an open porosity between 5 and 30%.

2. The filtration method according to claim 1, characterized in that the residence time of the liquid metal in the particulate material bed is greater than 1 sec and less than 500 secs.

3. The filtration method according to any of claims 1 or 2, characterized in that the porosity substantially stems from pores with a diameter greater than 10 μm and preferably between 10 and 200 μm .

4. The filtration method according to any of claims 1 to 3, characterized in that the material has a particle size between 0.2 and 20 mm and the bed has a thickness between 4 and 40 cm.

5. The filtration method according to any of claims 1 to 4, characterized in that the material is electrofused corundum.

6. The filtration method according to any of claims 1 to 5, characterized in that the liquid metal is selected from aluminium, magnesium or their alloys.

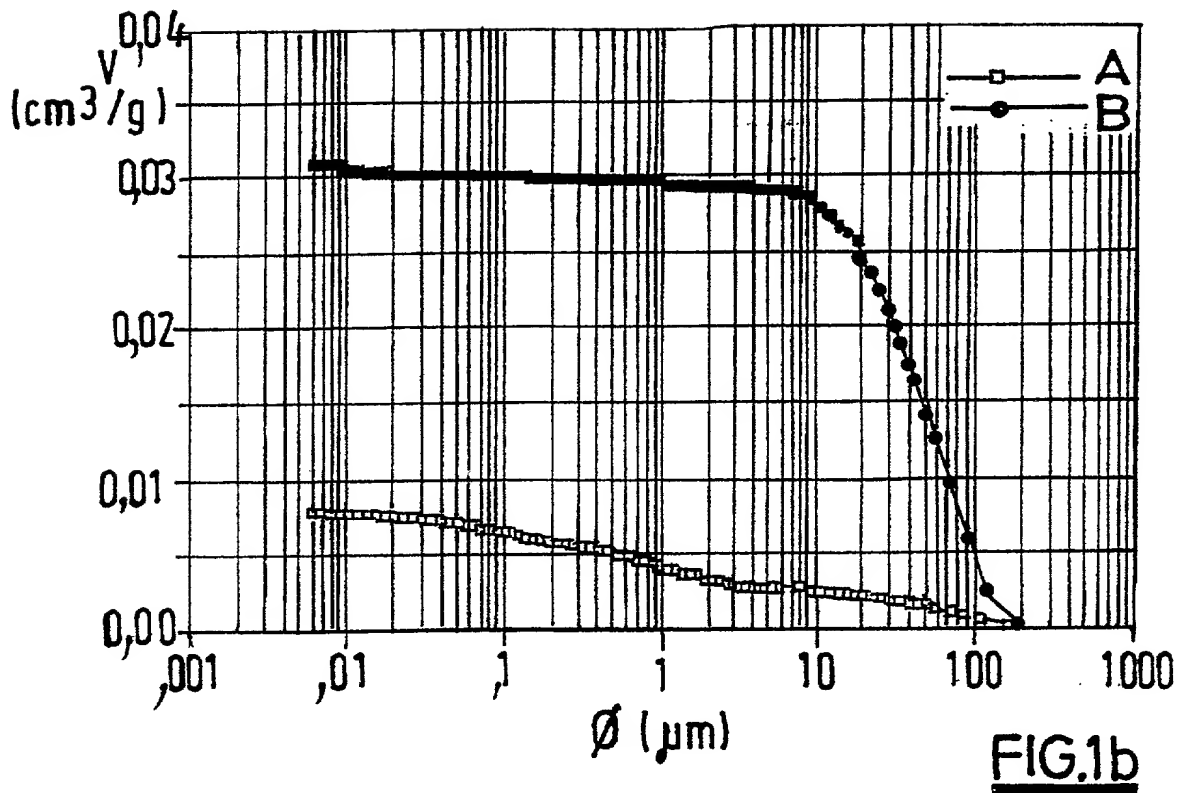
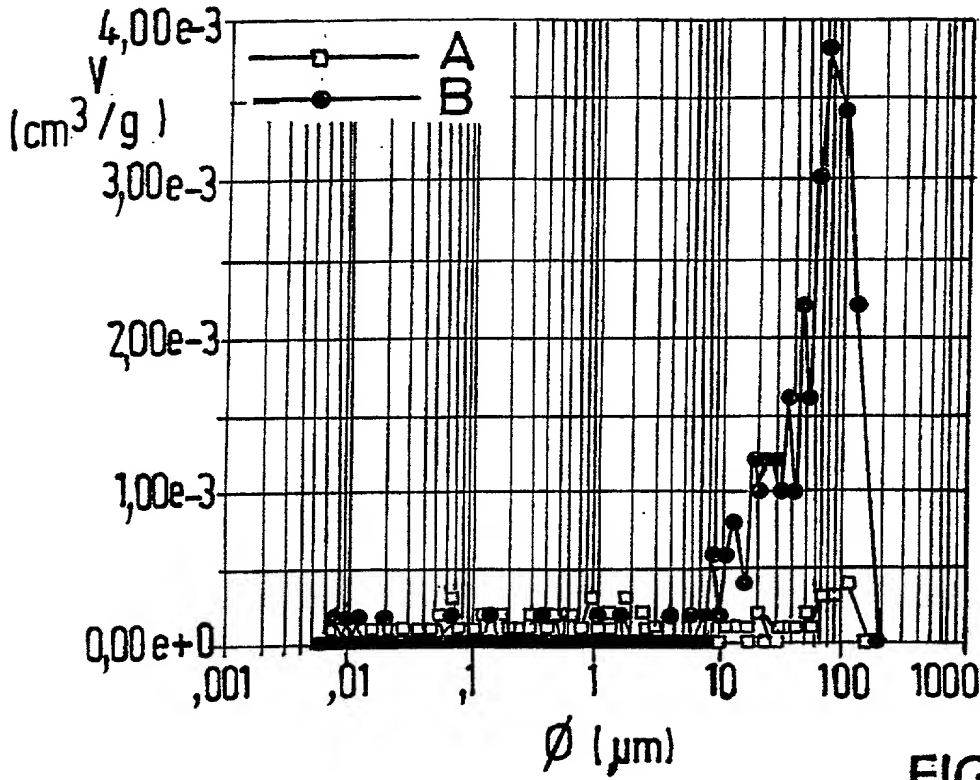
7. The method for obtaining corundum according to claim 5, characterized in that it comprises electrofusion of alumina, a casting, a cooling and solidification process in order to obtain said porosity, a crushing then a screening process.

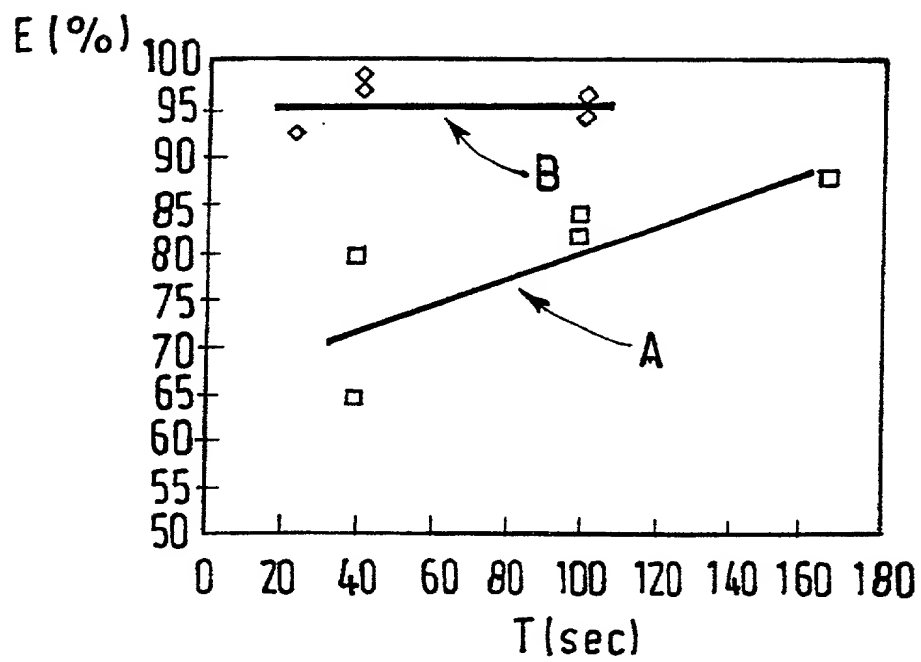
8. A corundum used in the method according to any of claims 1 to 6, or obtained according to the method of claim 7, characterized in that it has a porosity

between 5 and 30%.

9. A filtration device for liquid metal including the material according to claim 8.

10. Use of a filtration device for liquid metal
5 including the material according to claim 8 in the method according to any of claims 1 to 6.



FIG.2

DECLARATION FOR UTILITY OR DESIGN PATENT APPLICATION

Docket No. 01115

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled

IMPROVED METHOD FOR FILTERING A METAL LIQUID ON A BED OF REFRACTORY PARTICULATE MATERIAL

specification of which

(check one) _____ is described and claimed in PCT International Application

filed on (MM/DD/YYYY) _____ amended on _____

(if applicable)

(OR) XX is described in United States Application Number 09/856,460filed on (MM/DD/YYYY) June 7, 2001 (OR) _____ is attached hereto.

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in 37 CFR §1.56.

☒ I hereby claim foreign priority benefits under 35 U.S.C. §119(a)-(d) of any foreign application(s) for patent or inventor's certificate, or 365(a) of any PCT international application which designated at least one country other than the United States of America, listed below and have also identified below, any foreign application for patent or inventor's certificate, or any PCT international application having a filing date before that of the application on which priority is claimed.

Prior Foreign Application Number(s)	Country	Foreign Filing Date (MM/DD/YYYY)	Priority Claimed? Yes No
98 16389	France	December 21, 1998	XX
PCT/FR99/03184	PCT	December 17, 1999	XX

I hereby claim the benefit under 35 U.S.C. 119(e) of any United States Provisional Application(s) listed below.

I hereby claim the benefit under 35 U.S.C. §120 of any United States application(s), or 365(c) of any PCT International application designating the United States of America, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of 35 U.S.C. §112, I acknowledge the duty to disclose information which is material to patentability as defined in 37 CFR §1.56 which became available between the filing date of the prior application and the national or PCT international filing date of this application:

As a named inventor, I hereby appoint the following registered practitioner(s) to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith:

Donald L. Dennison
Burton Scheiner
Ira J. Schultz

Reg. No. 19920
Reg. No. 24018
Reg. 28666

Scott T. Wakeman Reg. No. 37750
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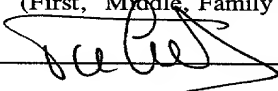
I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under 18 U.S.C. §1001 and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Full name of sole or first inventor Herve LESCUYER
(First, Middle, Family Name or Surname)

Inventor's signature [Signature] Date July 11th 2001

Residence Hermillon, France FRX Citizenship France
(City, State, Country)

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2-00
Full name of second joint inventor Alain DUBUS
(First, Middle, Family Name or Surname)
Second inventor's signature  Date July 25, 2001
Residence Biesheim, France FRX Citizenship France
(City, State, Country)
Full Post Office Address 11, rue du Saumon, 68600 Biesheim, France

Full name of third joint inventor _____
(First, Middle, Family Name or Surname)
Third inventor's signature _____ Date _____
Residence _____ Citizenship _____
(City, State, Country)
Full Post Office Address _____

Full name of fourth joint inventor _____
(First, Middle, Family Name or Surname)
Fourth inventor's signature _____ Date _____
Residence _____ Citizenship _____
(City, State, Country)
Full Post Office Address _____

Full name of fifth joint inventor _____
(First, Middle, Family Name or Surname)
Fifth inventor's signature _____ Date _____
Residence _____ Citizenship _____
(City, State, Country)
Full Post Office Address _____